BL (glyoxal) 6:30 a.m. 0.130

The "high moisture" tensile strength tests of the same products gave the following average values:

File

MINUTES

SUPERVISORS' MEETING

April 11-12, 1957

Present: A.E. O'Keeffe
J.C. Holmes
P.L. Gager, Jr.
F.A. Morrell
S.W. Fleasants (Chairman)
R.D. Hellams
C.C. Cosby, Jr.
J.D. Hind
J.Y. Mason, Jr. (Secretary)

ADMINISTRATIVE SESSION

Camp Kentwood, Beaufont Springs, and Hanover Wayside are being investigated as possible locations for a picnic.

Vacation schedules are due by April 25.

TECHNICAL SESSION

Project Reports

1-001C Gas Chromatography (Murrill, Gager)

Large glass wool filters remove almost all the particulate phase of cigarette smoke. When the gas phase is warmed after passage through such a filter, little or no tar is found in the trap. This gas phase of smoke will be checked for the presence of small amounts of furfural and butanedione.

The criteria of ultraviolet absorption values and chromatographic peak areas were used to show poor reproducibility in introducing small chromatographic samples (1 to 4 microliters). Replacement of the microsyringe barrel appears to give better results. Fraction trapping and transfer efficiencies will be checked under high flow rate conditions at 100°C.

1-002F Determination of Polyhydric Alcohols (Martin, Carpenter)

Recoveries of known glycerine by vacuum steam distillation average 80% when 50 mg. is cased on 2 grams of tobacco (uncased blend). Quantitative recoveries are made from glass wool. With the same amount of glycerine on 4 grams of uncased tobacco, only 70% of the glycerine is recovered. This is contrary to previous reports. Apparently, the glycerine reacts with some constituent of the tobacco.

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Source: https://www.industrydocuments.ucsf.edu/docs/tjxk0000

required for washing was about /-1/2 hours compared to 4 to 5 hours with a 40 mesh screen.

Production personnel are being trained so that they can take over the operation of the pulping equipment in a week or two.

Higher recoveries of glycerine were made from an all-tobacco binder (ATBL #68) than from CMC binder. The low recovery from CMC binder is probably due to esterification.

Water extraction followed by vacuum steam distillation has yielded a higher recovery of glycerine from laboratory cased tobacco than vacuum steam distillation alone. One, two, and four gram samples of tobacco have been cased with 50 mg. of glycerine. Glycerine determinations will be compared using water extraction plus vacuum steam distillation and vacuum steam distillation directly.

Increasing the moisture content of uncased tobacco (to approximately 20%) did not affect the recovery of glycerine.

1-003E Instrumental Research with the Mass Spectrometer
(Varsel, Bell, Resnik, Harrow)

Samples of the petroleum ether extracts of Turkish and bright tobaccos (petroleum ether extracts sprayed on glass wool) were collected and analyzed mass spectrometrically. Interpretation of results is not complete as yet.

Samples from the petroleum ether extracts of Coker 139 and Hicks tobacco varieties sprayed on glass wool were analyzed mass spectrometrically. Both were found to contain acetone and methanol along with traces of other organic constituents. The Coker 139 contained more acetone and less methanol than the Hicks. Other slight differences in peak ratios were noted.

After these samples had remained in the evacuated traps for two days a re-analysis showed an increase in nitrogen, CO_2, and argon in both cases. Apparently, the oxygen from the air was used up to form CO and CO_2, thus leaving an excess of N_2, argon, and CO_2 (above the amounts present in air).

Samples of toluene were analyzed quantitatively for the gas chromatography group to check percent recovery of samples.

The isatron filament burned out and instead of replacing the filament the schedule was moved ahead to permit installation of the new H.R. analyzer assembly. This assembly is presently being installed.

1-003E Instrumental Research by the Electronics Group (Morrell, Harrow)

The separate units of the fluorescence modification are being aligned and permanently anchored. This will complete the project except for future service work.

Parts are being assembled for an electrometer amplifier which will be used at the output of the ionization gauge to match the gauge to a recorder. A sketch of a modified ionization gauge is being submitted to Vacuum Electronics Engineering Corp. for a quotation on the construction of this gauge.

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c. Total Alkaloids: The results of analysis by the Griffith steam distillation procedure are in line with the perchloric acid procedure when the standard cell is used. We are now using a fast flow cell on the Cary spectrophotometer and a standard is in progress of being run to determine a factor.

Work on the platinum resistance thermal conductivity cell has been discontinued until a newly designed cell can be made. This cell uses capillary glass tubing throughout so as to eliminate all unnecessary dead space.

The new, high resolution analyzer assembly is being installed in the mass spectrometer. The installation is expected to be completed and checked out by April 15.

1-003F Absorption Spectroscopy (Bill, Resnik, Harrow)

Known reference spectra have been run and we are presently making a correct file of known reference spectra. One will be placed in alphabetical order, the other will be arranged according to types of compounds such as acids, etc.

Triacetin was identified and flavoring materials tentatively identified in the Newport filter plug.

Work has been started on the determination of menthol in the presence of triacetin in filter plugs. It has been found that carbon disulfide is the best solvent to use. Work will be done to determine the lowest concentration of menthol in the presence of triacetin that can be detected. (There is approximately 14 times more triacetin than menthol.) The variable path cell will be tried to compensate for triacetin in the sample and less CS_2 will be used to extract plugs.

1-003K Smoke Comparison with the Mass Spectrometer (Varsel, Bell, Resnik, Harrow)

No work has been done on this project during the past two weeks because of a lack of CO gas which is used as a reference standard.

2-001 Aging of Tobacco (Crayton, Mason)

Samples of well aged flue-cured tobacco, 5 years, were taken from the blending line. The very large saponification and ester numbers of this tobacco indicate that this phase of the aging process is continuous. Similar studies are being made on unaged controls and tobacco aged naturally for two and three years.

The aging of the petroleum ether extract of air cured tobacco on glass wool is continuing.

In addition to other differences, the mass spectrometric analysis of the unaged and aged ether extract of flue-cured tobacco showed the presence of acetone. This is in agreement with recently reported data of Japanese workers. Further investigations by mass spectrometric analyses are planned.

Steam distillation of the ether extract and the separation of the acid fraction of the volatile material will be attempted. The identification of the volatile acids could be most important in both the aging and flavor and aroma studies.

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2-001B-1 Determination of Carbohydrates in Tobacco (Greene)

An aqueous extract of stems was precipitated with methanol, and the residue hydrolyzed with acid. Paper chromatography indicates the presence of a pentose, probably resulting from the hydrolysis of pentosans.

2-001B-2 Determination of Alkaloids in Tobacco (Edmonds, Cager)

The benzene-chloroform extracts of tobacco used for the determination of alkaloids by perchloric acid titration will be used for paper chromatographic studies of alkaloids. When aliquots from these extracts were transferred to 0.25N HCl, excellent ultraviolet spectra were obtained. No substances should be present to interfere with the combined chromatographic-spectrophotometric method. Soxhlet extractions are being made to check the completeness of extraction by the shaking method.

2-001C Chemical Studies of the Petroleum Ether Extract of Tobacco (Carpenter)

A method is being developed to determine the glyceride content of the petroleum ether extract of tobacco. The neutral saponified extract is made acidic with aqueous acid and is extracted with ether. The aqueous layer is made to volume and the glycerine content determined by the chromatropic acid method.

2-003C Equilibrium Moisture Studies (Sharp, Mason)

The phosphate humectants described in the Ericsson patent showed no promise as satisfactory humectants. This study has been completed.

2-005 Evaluation of Tobacco (Bass, Sharp, Mason)

Tobacco Preservatives: No new results were obtained. A second proposed preservative from the Charles Pfizer Co. will be evaluated upon its receipt.

Enzyme Studies: Studies to determine if nicotine inactivates the enzymes are under way. Results from the initial determination of conversion of starch solutions containing 0, 0.02, 0.2, and 2 grams of nicotine, indicate that nicotine inhibits the conversion of starch to sugar but does not completely inactivate the enzymes. This experiment is being repeated and conclusions will be withheld until additional results are available.

Aroma and Flavor Studies: Mass spectrometric analyses of the petroleum ether extracts of Coker 139 and Hicks tobaccos showed differences between the two varieties. Coker 139 appeared to contain more acetone and less methanol than the Hicks tobacco. This study will be continued.

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Routine experimental work was halted to examine some specially prepared Spud filler (for English export). Both the unflavored filler and the menthol-treated filler showed a "glycerine" content of less than 0.10% by the chromatropic acid procedure. (British Customs allows up to 0.04% "flavor".)

Irrigation and Plant Population Studies: The results from the chemical analysis of samples representing these studies have been received and are being evaluated.

2-006B-1 Binder Development Studies (Estes, Clarke, Hellams, Butler)

This report period covers the following work:

1. Comparative work on paper chromatographic methods using samples obtained through various types of carbon columns. Results are not complete at this time.

2. All-tobacco BL composite representative samples of the fines portion of the ATBL have been obtained for exploratory work.

3. Reproducibility was determined on ATBL as prepared by our method. On twenty-three individually prepared samples, using the same ingredients on each, the following ranges were calculated at a 95% confidence level.

A. Tensile Strength

1) Against the grain = 0.675 to 1.271 kg/inch

2) With the grain = 0.565 to 1.207 kg/inch

Average of 1) and 2) = 0.627 to 1.233 kg/inch

B. Weight = 14.62 to 16.82 grams per sq. ft.

C. Tensile coefficient = 0.042 to 0.076 $\frac{\text{kg/inch}}{\text{gm/sq.ft.}}$

4. Samples of paper and ATBL will be compiled in the form of a ledger.

5. Microscopic investigation of pulp refining for the present BL at the BL plant is being completed and pictures will be made of the slides used for comparative work with the refined Herty pulps.

6. A detailed report of cold base soaking is being circulated.

7. The equipment for the cold base soaking under pressure is being ordered.

8. Investigation of the phosphate buffer digestion of stems is being completed at this time.

9. The equipment for the pentosan determination has been received. This analysis will be completed when time permits.

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Equipment suitable for extracting large quantities of tobacco (15 - 25 lbs.) is being investigated. This seems necessary because of the small quantities of some of the more interesting constituents.

10. Analysis of Doctor Knife Scrappings (DKS):

pH of BL = 4.60 pH of DKS = 4.50

	BL	DKS
Nicotine	1.23%	1.11%
T.V.B.	0.30%	0.29%
Total Nitrogen	1.91%	1.94%
Petroleum Ether Extract	3.6%	5.3%
Sugar	4.9%	3.1%
Total Base (milliequivalents per gram)	4.7	8.1
Total Acid (Milliequivalents per gram)	3.6	2.4

The chemical analyses indicate that DKS is similar to BL.

2-006B-2 Physical Testing of BL Products (Stewart, Cosby)

The physical testing of eleven samples of BL produced at the new plant has been completed. The data have been reported to Mr. Long's section for evaluation.

2-006B-2&3 Chemical, Physical & Smoking Properties of BL (Sharp, Mason)

No major difference in smoking characteristics has been observed between freshly prepared and the control BL.

2-006B-4 Sieve Analysis on BL Raw Materials (Stewart, Cosby)

Sieve analyses and sand analyses on each fraction of the sieved material are now in process on 18 samples received from the Production Department.

2-006B-4 Sand Analysis of BL (Pleasants, Wiley)

Determinations were made on 176 sand samples during this period.

Mr. Hind suggested that sand determinations be included in the quality control tests on finished BL.

2-006B-4 Binder Studies (Hind)

Titanium Dioxide as a Binder "Tag"

The most satisfactory method for handling samples containing TiO_2 is as follows:

1. Sample is placed in a "Vycor" crucible, heated on a hot plate, until completely charred.

2. One ml of H_2SO_4 is added to "sulfate" this ash, and the sample is placed in a muffle furnace set to 650°C to complete the disintegration, prior to dissolution in concentrated sulfuric acid. Spectroscopic standards previously obtained for Kjeldahl

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digestion procedures are being checked to see if they must be revised to conform with this method of preparing the samples.

Blank runs on tobacco dust, and several samples of current BL product show that a small but uniform correction to the spectrophotometric data may have to be applied.

Up to the present time, we have tagged at .4% based on binder solids. Since this is only 0.2% of BL weight, this amount may have to be raised.

Glutaraldehyde was tested in binder films from Herty F pulp and CMC. In binders of pH 4.5 (i.e. the pH of BL) no measurable wet strength could be observed even though glutaraldehyde was used at 0.3 and 0.6 pt. per pt. pulp.

However, in Guar gum binders, measurable wet strength of about 12% of dry strength was observed even at pH 7.5 when only 0.1 pt. glutaraldehyde per pt. pulp was used. At pH 3.0 to 3.5, wet strength was much higher, 46 to 50% of dry strength. Wet strength of Guar binder films at pH 4.5 is being determined for a wide range of glutaraldehyde content.

A procedure has been devised for measuring wet strength of BL, and is being used for a daily control of the product. The wet strength of present BL has varied over a wide range. Wet tensile strengths of a few Brooklyn pilot plant sheets have been measured and were found to be in the range of the present results.

4-002 Comparison of Brands (Sowell, Cosby)

On April 1st, the Comparison of Brands cigarettes were purchased from Cliff Weil Wholesale Tobacco Co., Richmond, Va. The physical and chemical testing of these cigarettes are progressing satisfactorily.

4-003A Leaf Analysis (Pleasants, Wiley)

The following analyses were completed on leaf and filler:

156 nicotine	280 petroleum ether extract
160 total ash	92 total nitrogen
91 T.V.B.	151 sugar
282 moisture	

total - 1412

4-003D Miscellaneous Laboratory Tests (Clarke, Estes, Hellams, Butler)

1. Moisture in Tobacco

The evaluation of this method will continue after arrival of the necessary equipment for handling a ten gram sample of filler tobacco. A statistical test will then be arranged to determine the precision of the method for the ten gram samples of filler tobacco.

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2. Total Acids in Cigarette Smoke

The work on this project has been side-stepped during this period in order to work on the following.

A. Beer's Law graph for furfural using the Klett colorimeter

B. Beer's Law graph for furfural using the Cary spectrophotometer.

C. Analysis of doctor knife scrapings (DKS) for:

1) Total acids = 2.4 milliequivalents of acid per gram of DKS.
2) Total bases = 8.1 milliequivalents of base per gram of DKS.

D. Analysis of BL for:

1) Total acids = 3.6 milliequivalents of acid per gram of BL.
2) Total bases = 4.7 milliequivalents of base per gram of BL.

3. Determination of Menthol in Cigarettes

A study is being conducted on the extraction step of the method. Extraction by shaking is being compared with elution.

4. Determination of Carbonyls in Smoke by 2,4-DNP's

2,4-DNP derivatives of DKS and BL were prepared. Spectrophotometric analysis showed the derivatives were dissimilar to each other and to any previous known sample for which we have reference spectra.

4-003D Miscellaneous Laboratory Tests (Pleasants, Wiley)

1. Plant Audit of Manufactured Cigarettes: The audit of fresh cigarettes from the Louisville and Richmond plants started in January, 1957, is current with the routine pickups.

To date the chemical analyses have shown no significant differences between samples of the same brand. The analyses run are nicotine, petroleum ether extract, total ash, total nitrogen, and reducing sugars.

2. NaOH in Sugar Solutions: No work was done on this project during this period.

3. Foil Separations: We continued the use of .1N nitric acid for the separation of foil from the paper liner. More work will have to be done on the solubility factor for aluminum in .1N nitric acid. Work will be resumed at a later date on the determination of aluminum by the 8-hydroxyquinoline procedure to check our present procedure.

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Mr. O'Keeffe suggested that the quality control determinations be made at the Reynolds plant. The paper and foil thickness could thus be determined before lamination and the adhesive thickness determined by the difference.

4. RKS in the Comparison of Brands: Cigarettes from the Richmond pickup of the April 1957 Comparison of Brands are being checked for RKS. The RKS is estimated at less than 1% or more than 1% of the filler.

5. Reconstituted Tobacco in the Comparison of Brands: To date, in the cigarettes from the Comparison of Brands that have been checked for reconstituted tobacco, it has been found in the following brands: Salem, Winston, Chesterfield, L&M, and Camels.

6. Total Alkaloids Steam Distillation Equipment: We have started using one station of the six station Griffith total alkaloid steam distillation unit. Insufficient samples have been run to determine its speed or accuracy. Its rated capacity is large.

4-004 Smoking Machines (Sowell, Cosby)

The 20 port smoking machine was operated on routine analyses during this period and the following smoke analyses were completed: 64 tar analyses; 104 nicotine analyses; 68 furfural analyses.

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